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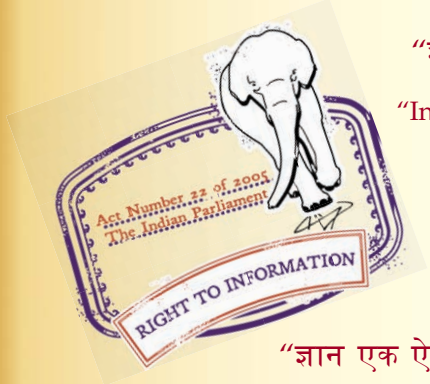
IS 3421 (1988): Textiles - Binary mixtures of acrylic, certain modacrylics and certain other fibres - Methods for quantitative chemical analysis [TXD 5: Chemical Methods of Test]



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"Knowledge is such a treasure which cannot be stolen"

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Indian Standard

TEXTILES — BINARY MIXTURES OF ACRYLIC, CERTAIN MODACRYLICS AND CERTAIN OTHER FIBRES — METHODS FOR QUANTITATIVE CHEMICAL ANALYSIS

(First Revision)

UDC 677-489'464 : 677'014'6

DESCRIPTORS : TEXTILES. BINARY FIBRE MIXTURES. SECONDARY CELLULOSE ACETATE AND CERTAIN OTHER FIBRES. CHEMICAL TESTS. QUANTITATIVE ANALYSIS.

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110 002

June 1989

Price Group 2

Chemical Methods of Test Sectional Committee, TDC 5

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 30 December 1988, after the draft finalized by the Chemical Methods of Test Sectional Committee had been approved by the Textile Division Council.

This Indian Standard was published in 1966 and has been revised to include another method based on dissolution of acrylic fibres in 80 percent (*m/m*) sulphuric acid, developed by the Textiles Committee, Bombay. This new method is less hazardous and more economical as compared to use of toxic and costly dimethyl formamide.

The use of different fibres blends in textiles has necessitated the formulation of standard methods for identification and quantitative estimation of respective fibres. The quantitative analysis of textile fibres in mixtures is of considerable importance to the textile technologists, traders and consumers.

While preparing this standard, considerable assistance has been derived from ISO 1833 : 1977 'Textiles — Binary fibre mixtures — Quantitative chemical analysis', issued by the International Organization for Standardization (ISO).

In reporting the result a test or analysis made in accordance with this standard, if the final value, observed or calculated, i be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical s (*revised*)'.

AMENDMENT NO. 1 FEBRUARY 1992
TO
IS 3421 : 1988 TEXTILES — BINARY MIXTURES OF ACRYLIC, CERTAIN MODACRYLICS AND CERTAIN OTHER FIBRES — METHODS FOR QUANTITATIVE CHEMICAL ANALYSIS

(First Revision)

(This amendment is being issued to remove any ambiguity regarding values of commercial moisture regain of different fibres to be taken into consideration at the time of calculating the test results.)

(*Page* 3, *clause* **8.3.2**, *Note* 1) — Substitute the following for the existing Note :

"For the purpose of calculations the commercial moisture regain values for various fibres as specified in IS 13157 : 1991 'Textiles fibres — Commercial moisture regains — Specification' shall be used."

(*Page* 3, *clause* **8.3.2**, *Note* 2) — Delete.

(TXD 5)

Reprography Unit, BIS, New Delhi, India

Indian Standard

TEXTILES — BINARY MIXTURES OF ACRYLIC, CERTAIN MODACRYLICS AND CERTAIN OTHER FIBRES — METHODS FOR QUANTITATIVE CHEMICAL ANALYSIS

(First Revision)

1 SCOPE

1.1 This standard prescribes two methods for the quantitative chemical analysis of binary mixtures of acrylic, modacrylic fibres with wool, silk, cotton, polyamide, polyester, viscose, cupro, modal or glass fibres. It is suitable for application to fibres in any textile form, such as fibre, yarn or fabric. Method 2 is not applicable to acrylic fibres containing cellulosic fibres, silk and nylon (polyamide).

1.1.1 It is applicable to acrylic fibres dyed with premetallized dyes but not to those dyed with after chrome dyes. It covers only those modacrylic fibres which are completely soluble in dimethyl formamide (DMF).

NOTE — Before conducting an analysis according to this standard, the fibres present in the mixture should be identified (*see* IS 667 : 1981) and the sample to be analysed should be freed from all non-fibrous matter (*see* IS 9068 : 1979). Dye in the dyed fibre is considered to be an integral part of the fibre and is not to be removed.

2 REFERENCES

The Indian Standards listed below are necessary adjucts to this standard:

IS 667 : 1981	Methods for identification of textile fibres (*first revision*)
IS 1070 : 1977	Specification for water for general laboratory use (*second revision*)
IS 9068 : 1979	Recommended methods for the removal of non-fibrous matter prior to quantitative analysis of fibre mixtures

3 SAMPLING

3.1 Lot

The quantity of textile material of one definite type and quality delivered to a buyer against one despatch note shall constitute a lot.

3.1.1 If the textile material is fibre or yarn and the lot consists of more than 200 kg of fibre or yarn, it shall be divided into sub-lots, each weighing 200 kg or less.

3.1.2 Each sub-lot shall be tested separately.

3.2 Sampling for Fibre and Yarn

From a sub-lot 15 increments, each approximately weighing 10 g, shall be taken from different parts and mixed thoroughly. This shall constitute a test sample.

3.3 Sampling for Fabrics

3.3.1 The number of pieces to be selected shall be in accordance with Table 1. The pieces thus selected shall constitute a gross sample.

Table 1 Sample Size
(*Clause* 3.3.1)

Lot Size (Number of Pieces)	Sample Size (Number of Pieces)
Up to 100	3
101 to 300	4
301 to 500	5
501 to above	7

3.3.2 From each piece in the gross sample selected as in **3.3.1**, cut out small portions from at least two different parts weighing about 25 g. The parts selected shall well represent the gross sample as far as possible. In the case of fabrics with a definite repetition in weave pattern, the parts selected shall include all yarns in the complete repeat. Dissect the small portions of fabric thus collected into yarn and mix them thoroughly. This shall constitute a test sample.

4 APPARATUS

4.1 Conical Flask

Of 200 ml minimum capacity, provided with a ground glass stopper.

4.2 Sintered Glass Filter Crucible

Of appropriate capacity with a pore size of 90 to 150 microns (porosity 1) and provided with a ground glass stopper. If stopper is not available, the crucible should be enclosed in weighing bottle for weighing.

4.3 Ventilated Oven

For drying samples at 105 ± 3°C.

4.4 Filter Flask

With connection to filter pump and adaptor to enable the crucible (*see* **4.2**) to be fitted to it.

4.5 Analytical Balance

Capable of weighing to an accuracy of 0·000 2 g.

4.6 Desiccator

Containing self-indicating silica gel or anhydrous calcium chloride.

4.7 Mechanical Shaker

5 REAGENTS

5.0 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (*see* IS 1070 : 1977) shall be used where the use of water as a reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

5.1 Dimethyl Formamide

Boiling point 152 to 154°C.

5.2 Sulphuric Acid Solution

80 percent (*m/m*).

5.3 Dilute Sulphuric Acid Solution

5 percent (*m/m*).

5.4 Dilute Ammonia Solution

Prepared by diluting 80 ml of concentrated ammonia solution (specific gravity 0·89) to one litre with distilled water.

6 TESTING CONDITIONS

The test shall be conducted in prevailing atmospheric conditions.

NOTE — Since dry masses are determined, it is not necessary to condition the sample.

7 PREPARATION OF TEST SPECIMENS

From the sample, after removing size and finishes as recommended in IS 9068 : 1979, draw a representative sample weighing about 2 to 3 g. Cut the yarn into pieces and dissect the cloth into yarn pieces of about 10 mm length.

8 METHOD 1

8.1 Principle

8.1.1 A sample of the mixture is dried and weighed. The acrylic or modacrylic fibres in the mixture are dissolved in dimethyl formamide at 90 to 95°C. The residue, that is, insoluble component, is collected, washed, dried and weighed; its mass corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of acrylic or modacrylic is found by difference.

8.2 Procedure

8.2.1 Take a test specimen weighing about 1 g from the pretreated sample (*see* 7). Dry the specimen in a weighing bottle at 105±3°C to constant mass, cool it in a dessicator and obtain the oven dry mass of the specimen.

NOTE — The mass shall be taken as constant if the difference between any two successive weighings at an interval of 20 minutes does not exceed 0·1 percent.

8.2.2 Put all the pieces into a 200-ml conical flask, and add 80 ml of dimethyl formamide per gram of the specimen. Insert the stopper, shake the flask to wet out the specimen and heat for 10 min, at 90 to 95°C in a water bath. Shake gently the contents of the flask five times during this period. Decant the solution through the tared sintered glass filter crucible. Filter the contents of the flask through the filter crucible by means of suction. Add 60 ml of dimethyl formamide to the residue in the flask, shake by hand and decant the liquid through the filter crucible. Tansfer the fibres, remaining in the flask, to the crucible by washing out the flask with distilled water. Apply suction to the crucible to remove excess water. Wash the residue twice with water by filling the crucible, allowing it to drain under gravity and then draining with suction.

8.2.3 If the residue consists of polyamide or polyester, dry the crucible and residue at 105±3°C and cool and weigh them. If the residue is viscose rayon, cotton, silk or wool, transfer it with forceps to a 200 ml conical flask. Add 160 ml of distilled water and shake vigorously, intermittently for five minutes. Decant through the filter crucible and repeat the washing process three times more. After the last washing, filter the contents of the flask through the crucible by means of suction. Transfer the fibres remaining in the flask to the crucible by washing with distilled water and apply suction to the crucible. Dry the crucible and residue at 105±3°C, cool in a desiccator and weigh them. Determine the oven-dry mass of the residue.

8.2.4 Repeat the procedure prescribed in **8.2.1** to **8.2.3** with the remaining test specimen(s).

8.3 Calculations

8.3.1 *Method Based on Clean Dry Mass*

Calculate the percentage (P) of clean dry insoluble component by the formula:

$$P = \frac{100 \times m_1 \times d}{m_0}$$

where

m_0 = the dry mass of the specimen;

m_1 = the dry mass of the residue; and

d = the correction factor of variation in mass of the insoluble component in the reagent.

NOTE — Suitable values of d are as follows:

Fibre	*d*
Cotton	1·00
Nylon 6 or 6·6	1·01
Polyester	1·02
Silk	1·00
Wool	1·01
Viscose rayon, cupro, modal	1·01

8.3.2 *Method Based on Clean Dry Mass with Percentage Additions for Moisture*

Calculate the percentage (P_M) of clean insoluble component with percentage additions for moisture, by the formula:

$$P_M = \frac{100 \times P \times \left(1 + \frac{b}{100}\right)}{P\left(1 + \frac{b}{100}\right) + (100 - P)\left(1 + \frac{a}{100}\right)}$$

where

P = percentage of clean dry insoluble component,

a = percentage addition for moisture to the soluble component, and

b = percentage addition for moisture to the insoluble component.

NOTES

1 The following values for standard moisture regain of various fibres may be considered:

Fibre	*Standard Moisture Regain (Percent)*
Acrylic	1·50
Aramid (Safety apparel fabrics)	4·50
Cotton	8·50
Modacrylic	0·40
Nylon (polyamide)	4·50
Polyester	0·40
Silk	11·00
Textile glass	Zero
Viscose rayon, cupro, modal	13·00
Wool	13·60

2 The standard moisture regain values are generally accepted as the commercial moisture regain values in the trade.

8.3.3 *Method Based on Clean Dry Mass with Percentage Additions for Moisture and Non-fibrous Matter*

Calculate the percentage (P_A) of clean insoluble component in the mixture with percentage additions for moisture and non-fibrous matter by the following formula (*see also* Notes 1 and 2 under **8.3.2**):

$$P_A = \frac{100 \times P \times \left[1 + \frac{a_2 + b_2}{100}\right]}{P \times \left[1 + \frac{a_2 + b_2}{100}\right] + (100 - P)\left[1 + \frac{a_1 + b_1}{100}\right]}$$

where

P = percentage of clean dry insoluble component,

a_1 = percentage addition for moisture to the soluble component,

a_2 = percentage addition for moisture to the insoluble component,

b_1 = percentage addition for non-fibrous matter to the soluble component, and

b_2 = percentage addition for non-fibrous matter to the insoluble component.

NOTE — The percentage additions for non-fibrous matter may be as agreed to between the buyer and the seller.

8.4 Find out the percentage of second component by difference.

9 METHOD 2

9.1 Principle

9.1.1 A sample of the mixture is dried and weighed. The acrylic or modacrylic fibres in the mixture are dissolved in 80 percent (*m/m*) sulphuric acid solution at room temperature for 30 minutes. The residue, that is, the insoluble component, is collected, washed, dried and weighed; its mass corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of acetate is found by difference.

9.2 Procedure

9.2.1 Follow the procedure given in **8.2.1**.

9.2.2 Put all the pieces into a 200-ml conical flask and add 100 ml of 80 percent (*m/m*) sulphuric acid (specific gravity 1·725) per gram of the specimen. Insert the stopper, shake the flask to wet out the specimen and allow to stand at room temperature for 30 minutes shaking it at intervals. Filter the contents of the flask through a weighed filter crucible and transfer any residual fibres to the crucible by washing out the flask with a little more sulphuric acid. Drain the crucible with suction and wash the residue on the filter successively with dilute sulphuric acid (5 percent), hot water, dilute ammonia solution, and finally cold water, draining the crucible with suction after each addition. (Do not apply suction until each washing liquor has drained under gravity.)

9.2.3 Follow the procedure described in **8.2.3**.

9.2.4 Repeat the procedure given in **9.2.1** to **9.2.3** with the remaining test specimen(s).

9.3 Calculations

9.3.1 Calculate the percent of component fibres in the mixture as prescribed in **8.3.1**, **8.3.2**, **8.3.3** and **8.4** taking the value of correction factor d as 0·947 for wool and 1·00 for all other fibres.

10 REPORT

10.1 The report shall include the following:

a) Nature of material to be tested;

b) Method used (*see* **8** or **9**),

c) Method of calculation used (*see* **8.3** and **9.3**);

d) Number of specimens tested, and

e) The percentage of component fibres in the mixture (individual and average).

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